Dispersions of Ink-Jet Pigments: Using NMR Relaxation Measurements as a Quality Control Tool

Introduction

The application performance of any pigment is determined by its nature, including how it was manufactured, and the level of dispersion achieved in formulation. The most important physical properties include particle size and wetted surface area. The finer the particle size, the more intense will be the color; the greater the surface area, the greater will be the extent and uniformity of surface coating. For industries that must produce products with reliably consistent colors, measuring particle size and wetted surface area quickly and easily will aid in more efficient formulation and help to reduce production costs.

All dry powders will initially comprise primary particles, aggregates, and agglomerates, but it is the efficiency of the dispersion process that determines the final particle size distribution and, hence, the impact on total wetted surface area. Because high total surface area is important, aggregation in suspensions needs to be kept to a minimum, and any analytical technique that can provide an indication of the degree of aggregation is most welcome. The level of dispersion in any given formulation is, of course, strongly influenced by the mixing equipment and procedures used. However, owing to the opacity and high concentrations normally used, precise and accurate physical characterization of nanosize pigment dispersions is often difficult.

The most common method of surface area determination is nitrogen gas adsorption, but this technique is useful only for dry powders. It provides no information about the wetted surface area of suspensions (see Mageleka White Paper #1). Further, it requires extensive sample preparation (e.g., a sample must first be degassed) which can be very time consuming.

So, what technique can make fast, reliable, direct measurements of wetted surface area in any suspension and, particularly, nanosize pigment dispersions? Nuclear magnetic resonance (NMR) relaxation, which is the basis for Mageleka's *M*agno*M*eter XRS™, can directly measure the wetted surface area of any particulate suspension.

About NMR Relaxation

NMR spectroscopy is one of the most powerful analytical tools used to probe details of molecular structure and dynamics. Devices employing NMR technology require very high magnetic fields and, hence, very large magnets. However, the advent of small powerful magnets has allowed instruments - such as the Mageleka *M*agno*M*eter XRS™ - to be designed that are suited to normal, routine laboratory analysis.

The basic technique used in the *M*agno*M*eter is NMR relaxation. The relaxation time is a fundamental intrinsic property of solids and liquids and its measurement provides direct information about the extent and nature of any particle-liquid interface (i.e., suspensions and emulsions; see Mageleka Technical Note 1).

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What does the *MagnoM*eter do?**

The *Magno**M*eter provides complementary information and intelligence to traditional particle characterization devices. As mentioned above, the basic measurement is a relaxation time which is directly proportional to the wetted surface area of the suspension or slurry. The calculation of a surface area value is quite straightforward (see Mageleka White Paper 1). This is in contrast to the measurement of particle size by light scattering where the raw intensity data has to be deconvoluted by means of complex algorithms, and the assumption of particle sphericity must be made.

The actual relaxation value obtained by NMR is an average, and is dependent upon the exact composition of the suspension. This is somewhat analogous to the zeta potential of a material where the value depends critically upon the exact composition of the dispersion fluid. The surface area value calculated from NMR relaxation is based on a calibration using a dispersion of the same type of particles where the surface area is already known (see Mageleka White Paper 1).

The *Magno**M*eter's measurement technique is non-invasive and non-destructive and the *Magno**M*eter can work with suspensions at any industrially-relevant concentration. This is especially important when working with pigment dispersions. Importantly, the *Magno**M*eter eliminates the dilution issues inherent in making measurements using, for example, traditional light scattering techniques. Moreover, the simple measurement technique takes only minutes (see Mageleka Technical Note 2: The Mageleka *Magno**M*eter: What is it and Why use it?)

Utilizing Wetted Surface Area Measurements for Quality Control of Ink-Jet Pigments

In this case study, aqueous dispersions of various ink-jet pigments were supplied by a major manufacturer. They were yellow, magenta, and cyan (Table 1), and the materials in question were considered to be standards. For the yellows, two different primary particle size pigments were tested. For any suspension, the relaxation time decreases with increase in concentration. Hence, in this example, since the percent solids was not quite equivalent for each pigment "pair", in the calculation of surface area for them, the raw data was normalized with respect to concentration. Each set comprised batches that had been subjected to the same processing but were

Table 1. Surface area values in relation to hiding power of sets of dispersions formulated using yellow, magenta, and cyan pigments.

Pigment	Solids (wt%)	Surface Area (m^2g^{-1})	Hiding Power* Pass/Fail
Y1A	12.9	83	Y
Y1B	13.4	60	N
Y4C	10.8	30	Y
Y4D	10.0	26	N
M1A	18.3	90	Y
M1B	18.5	31	N
C1E	15.6	56	Y
C1F	15.3	46	N

formulated with different polymeric dispersing aids. For each pigment pair (i.e., A&B, C&D, etc.), two different polymers were used (i.e., one for A and a different one for B, etc.). Each suspension was tested for hiding power using a procedure adapted from the American Society for Testing of Materials (ASTM) standard method for determining the hiding power of paints.

The data in Table 1 summarizes the surface area values calculated from measurements of NMR relaxation time.

As might be expected, the data correlates well with pass/fail criteria for hiding power: the greater the surface area, the better the hiding power. It is also clear that relaxation measurements can discriminate not only between materials of different particle size but also between the efficacies of different polymeric dispersing aids.

The speed and simplicity of NMR-based measurements with the *M*agno*M*eter make it an ideal tool for routine quality control purposes. The advantage of such measurements is that important characteristics of products, from fundamentals to end-use performance, can be monitored quickly and easily – and at virtually any industrially relevant solids concentration – thereby saving time and money.

For more information, to send samples, to arrange a demonstration of the MagnoMeter at your facility, or to talk to one of Mageleka's technical applications specialists, please email roger@mageleka.com

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